Naval Research Laboratory
Washington, DC 20375-5320

NRL/MR/6930--97-7931

Certain Properties of Laboratory Greywater and Shipboard Non-Oily Wastewater and Permeates

DAN ZABETAKIS
BRUCE PAUL GABER

Center for Bio/Molecular Science and Engineering

May 21, 1997

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY (*Leave Blank*)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	May 21, 1997	

4. TITLE AND SUBTITLE

Certain Properties of Laboratory Greywater and Shipboard Non-oily Wastewater and Permeates

5. FUNDING NUMBERS

6. AUTHOR(S)

Dan Zabetakis and Bruce Paul Gaber

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

Naval Research Laboratory
Washington, DC 20375-5320

8. PERFORMING ORGANIZATION REPORT NUMBER

NRL/MR/6930--97-7931

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

Naval Surface Weapons Center
Carderock Division
Bethesda, MD 20084-5000

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT

Approved for public release; distribution unlimited.

12b. DISTRIBUTION CODE

13. ABSTRACT (*Maximum 200 words*)

Waste water samples were collected from experimental greywater treatment filters. Greywater source was from the U.S. Naval Academy, Annapolis, MD; from DD977, USS Hayler, or from CG51, USS Thomas S. Gates, stationed at Norfolk, VA. The waste was analyzed for general and specific properties and constituents. The total dissolved solids ranged from 546 mg/l to 8140 mg/l, and the chemical oxygen demand from 122 mg/l to 1160 mg/l. The concentrations of protein, carbohydrate, surfactant, as well as organic and inorganic ions were highly variable, and showed no correlation between themselves or with the bulk characteristics. The molecular weight range of material passing through the filtration system was determined, revealing that the majority of material is very small, with greater than 50% passing an ultrafilter of 3000 molecular weight cutoff. The efficiency of the filtration process was examined, showing that about 60% of COD was removed, as well as essentially all of total suspended solids.

14. SUBJECT TERMS

Greywater
Filtration
Sewage
Wastewater

15. NUMBER OF PAGES

56

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	UL

NSN 7540-01-280-5500

Standard Form 298 (Rev. 2-89)
Prescribed by ANSI Std 239-18
298-102

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CERTAIN PROPERTIES OF LABORATORY GREYWATER AND SHIPBOARD NON-OILY WASTEWATER AND PERMEATES

INTRODUCTION

Shipboard liquid waste originating from showers, kitchens and laundry, known as greywater, is a current and future problem faced by the Navy(1). As this type of waste is far less dangerous and polluting as compared to sewage, previously greywater was disposed of overboard. Current Navy policy has led to the establishment of a goal for the reduction of greywater pollution to a biological oxygen demand (BOD) level of 5 mg/l. The Naval Surface Warfare Center, Carderock Division, in a program under the management of Joseph Pizzino (NAVSEA Code 03R16) is engaged in an effort to develop a filtration treatment system for use prior to disposal. Commercial filters are being tested for their ability and practicality to remove pollution-causing compounds. Our effort has been directed at analysis of the permeate liquid that has passed through this system.

Of the many characteristics of water, one of the most direct measures of water quality is oxygen demand, usually expressed as either BOD or chemical oxygen demand (COD). A major component of pollution entering a water environment is organic compounds that may be used as food by aquatic microorganisms. This is particularly true for greywater as food waste may be a major contributor. When microorganisms consume the food, they also consume dissolved oxygen from the water for use in respiration. As this dissolved oxygen is essential also for fish and other forms of life in the water, a significant addition of pollutants to water may lower the dissolved oxygen to levels that make the water uninhabitable. BOD and COD are measures of the oxygen consuming capacity of waste(2). Determination of BOD is accomplished by measuring the depletion of oxygen in a closed sample seeded with bacteria. COD is determined by measuring the chemical reduction of chromium by the sample.

Manuscript approved January 31, 1997

COD was chosen as the experimental technique for this study as it is simpler and easier to apply to waste samples of highly variable nature. The goal of the analysis is to identify those components of the greywater that contribute most to the COD, and to characterize them as much as practical. Further, a molecular weight characterization of the permeate was judged critical, as the greywater treatment system is filtration based.

A secondary goal of the project was to determine the variability of greywater and permeate composition. To this end, a broad range of assays for organic and inorganic components were conducted. This includes assays of bulk properties such as dissolved and suspended solids, as well as analysis of specific ions and compounds.

METHODS

Wastewater

Samples were provided by John Benson (NSWC Code 633). The origin of the greywater was from the U.S. Naval Academy, Annapolis, MD. Non-oily wastewater (a combination of sewage and greywater generated aboard ship) was from a pierside connection to DD997 or CG51 at Norfolk, VA. Table 1 lists the samples, the collection date, origin, and known source. Samples from Annapolis were transferred the same day to NRL in clean polypropylene containers. Samples from Norfolk were express mailed overnight to NRL frozen in glass or plastic bottles. Most samples did not contain observable (by microscopic examination) bacteria or other microbes when collected, but several did (including all of the feed samples). All samples contain some level of bacteria, and when left at room temperature for several day showed evidence of bacterial growth. All analyses were conducted as soon as possible after samples were acquired and none were attempted when significant bacterial growth was observed.

Chemical Oxygen Demand (COD)

The COD assay is based on a colorimetric reduction of chromium under acid conditions at high temperature. Standard low range COD test vials were purchased from Bioscience, Inc., and used according to manufacturer's instruction. For each sample, 2.5 ml was placed into the vial and capped. The vial was shaken and incubated for 2 h at 125°C. After cooling, the absorbance at 440 nm was measured. A series of standards was prepared from potassium hydrogen phthalate (Sigma) and used to calibrate COD. If necessary, out of range waste water was diluted and retested.

Total Dissolved Solids (TDS)

Total dissolved solids is measured by conductivity of aqueous solution in a standard electrode detector. Conductivity, resistivity, and TDS were measured directly in the waste water with a FisherBrand Conductivity Meter (Fisher). Temperature of the solution at time of measurement was also reported.

Total Suspended Solids (TSS)

Aliquots of waste water were taken of various sizes depending on the estimated amount of particulate matter in the sample (100ml to 1000ml). The sample was passed through a pre-weighed Gelman Type A/E Glass Binder-free Filter with a nominal 1 micron opening size. The filter was recovered and dried at 103°C and re-weighed. The difference was recorded and reported as mg/l.

Non-Volatile Material (NVM)

Aliquots of 250ml to 500ml were evaporated in a Rotavap concentrator until the total volume was reduced to approximately 20-30 ml. The solution was transfered to a pre-weighed

boat, and the flask washed. The flask and wash water was sonicated if necessary to remove dried material. The wash was added to the boat, and evaporated at 103°C. The difference in weight was reported as mg/l. This assay may also be termed Total Solids.

Extractable Material

A measured amount (usually 500ml) of sample was added to a separatory funnel. Three extractions of 100ml each were conducted, and the organic layer was collected. Methylene chloride was used for all organic extractions. The combined organic layers were evaporated and weighed as described for Non-Volatile Material. No difference was noted when the water sample was acidified with HCl prior to extraction.

Protein Determination

Total protein was determined by a standard test kit (Sigma Diagnostics) based on the Lowry dye-binding assay. A standard series of protein concentrations using bovine serum albumin was prepared for standardization. By comparison to the standard curve and a blank sample, total protein was determined for each sample. Results were converted to mg/l.

Carbohydrate Determination

Total carbohydrate was determined by reaction with the anthrone reagent(3). Anthrone reagent consists of 2 mg/ml anthrone (Aldrich) in 13.5M H_2SO_4. Waste water was added to test tubes (1 ml) and mixed with 5 ml anthrone reagent. Tubes were heated to 125°C for 10 minutes. Absorbance was measured at 625nm and compared to standard solutions of glucose to determine carbohydrate concentration in mg/l.

Filtration Experiments

To determine the amount of organic contaminants in wastewater, ultraviolet absorbance

was measured at both 205nm and 280nm. The higher wavelength is characteristic for the amino acids tryptophane and tyrosine, and can be used to estimate protein levels. However, other organic chemicals may also absorb at this wavelength. Not all organic compounds will absorb UV light, so no attempt at quantification was made.

The absorbance of sample prior to any filtration was determined. Occasionally, solid particulates were present in the samples. These particles were removed by brief centrifugation, and their contribution to the sample contents was neglected. An aliquot of 2.5ml was added to the upper chamber of a Centricon-3, -30, or -100 (Amicon) centrifuge ultrafilter. The filter was subjected to approximately 1000 X g in a C-6000 centrifuge (IEC, Inc.) for 5 to 15 minutes. The filtrate was collected and the UV absorbance was measured. The data was reported as a fraction or percent of the starting absorbance.

Gas Chromatography, Mass Spectrometry

Organic compounds were extracted from the waste water samples by one of two methods. The first was by multiple extractions with methylene chloride as describe above. The second method was by continuous liquid-liquid extraction in a modified Hershberg-Wolfe apparatus (Kontes). Both procedures were followed by evaporative concentration in a Kuderna-Danish apparatus (Kontes). Concentrates were then injected into a Hewlett Packard Model 5890 GC-MS for analysis. The temperature was raised from 40°C to 250°C over 20 minutes. Total ion count was used for quantification, and was reported in mg/l relative to a series of standards. Concentrations were not standardized against the chemicals identified, and should be used only as a rough guide to actual concentrations. Identification of specific chemicals where possible was done by comparison to the Wiley GC-MS database. Identifications where reported were not

followed up, are subject to error, and should only be relied on with caution.

Other Specific Assays

Specific assays for chloride, chlorine, phosphate, copper, surfactant, zinc, sulfide, hardness, nitrate, and lead were conducted according to manufacturer's instructions using standard commercially available test kits (Orbeco-Hellige).

RESULTS

Individual sample reports are presented in the Appendix.

Permeate constituents and variability

Wastewater experiments conducted by the Naval Surface Warfare Center involved the filtration of feed wastewater and the production of a permeate. Samples of permeate were collected and assayed to determine general properties and specific constituents. The results are summarized in Table 2. Gross properties for laboratory samples are summarized in Figure 1.

A major finding is the high degree of variability found for most properties and constituents. A further point is that there is no correlation between any pairs of data. The data cannot be explained in terms of some samples being simply more 'dirty' than others. Samples high in protein (5 and 11) may or may not be high in carbohydrate (5 and 11) or extractable material (3 and 5) or surfactant (4 and 5). Those sample highest in chloride (3, 4, and 7) have variable levels of protein, phosphate, nitrate, and surfactant. Similar results are apparent for any pairing of assays.

Samples 1 through 5 are from the U.S. Naval Academy at Annapolis, while samples 7 through 13 originate aboard ship. While the same conditions of variability are apparent in both sample sets, there are two differences to note between the sources. The pH of Annapolis samples

was within the range of 8.1 to 10.0, while the shipboard samples were acidic, ranging from 4.9 to 6.8. The shipboard samples also usually contained more total material (TDS, NVM), even though the assays for specific components did not correspondingly increase. The peak values for TDS was over 8 grams per liter, while the Annapolis samples were limited to about 1 gram per liter. This increase in total material does not correlate with the COD levels, which were highly variable.

Non-Volatile Material and Total Dissolved Solids are two measures that should be broadly proportional. Although the analytical technique for each is based on a different principle, in a mixed waste they could be expected to yield similar values, as is seen for these samples. Figure 2A shows a linear regression plot of NVM versus TDS. With a few notable exceptions, the two parameters are proportional, yielding a line with slope of 0.84 and a correlation coefficient of 0.87. A slope of 1 would mean direct 1:1 proportionality. Figure 2B shows a similar plot of NVM and TDS versus COD. No relationship is apparent, represented by a correlation coefficient of 0.0028.

Protein levels also show a lack of correlation with other variables. Figure 2C shows that protein levels are not proportional to COD or to TDS. The regression to COD has a slope of -0.04 and a correlation coefficient of 0.11, while TDS results are a slope of -0.00061, and a correlation coefficient of 0.00083.

Molecular weight characterization

The commercial filters used in the greywater treatment system have a nominal molecular weight limit of about 100,000 Daltons. Freshly filtered samples should have no molecules or aggregates larger than this cut off. Over time, however, waste material may flocculate, and

bacteria and other microorganisms may grow. To determine the molecular weight distribution of fresh samples, we conducted a series of filtrations with small scale centrifuge ultrafilters. An aliquot of 2.5 ml was subject to filtration at a cut off of 100,000 Daltons, 30,000 Daltons, and 3000 Daltons.

The analytical technique used to monitor general organic compounds was ultraviolet absorbance. The absorbance spectrum of greywater samples reveals no significant peaks, only a large broad increase in the lower UV range, and a detectable shoulder at approximately 280nm. As 280nm coincides with the absorbance peak of protein, this was chosen as a relevant wavelength for the assay. The wavelength of 205nm was chosen as representative for the lower UV range, as very many organic compounds have significant absorption in this region.

Figure 3 is a summary of the data for permeate samples 1 through 9. In all cases, greater than 50% of the UV absorbing material passed through even the 3000 Dalton filter, and typically only a maximum of 30% of material could be removed by this enhanced filtration.

Removal of constituents by filtration

Samples 7 through 14 are pairs of greywater feed and permeate. Data for gross constituents of the feed samples is presented in Figure 4. Where possible, results from the analysis were compared, and a percent removal was calculated. This data is summarized in Figure 5. The feed samples were considerably more 'dirty' than the permeate samples, contained large amounts of suspended matter, and often failed to yield readable results in the assays. Sample 7 permeate contained an exceptional amount of total dissolved solids. One possible explanation for this anomalous result is that the filtration system had been used on the previous day to filter excessively salty water. It is possible that the permeate when collected contained salt

that had been retained in the system, while the corresponding feed contained considerably less.

The results show that the filtration process is highly variable for removal of TDS and NVM, ranging between 95% and none. The filtration was, however, highly efficient at the removal of suspended solids (TSS), removing all or close to all of TSS present in the feed. COD removal was also less variable. About 60% of COD was removed in each of the four samples tested.

Gas chromatography

Waste water samples were extracted with methylene chloride, and concentrated approximately 300-fold. A portion of this extract was injected into a Hewlett Packard Model 5890 GC/MS. Figure 6 shows total ion count for two samples, one from Annapolis (sample 2), and one from Norfolk (sample 7). Although there is variability, samples 1-6 were similar to sample 2, while samples 7-14 resembled sample 7. Specific compounds identified by comparison to the Wiley database are indicated, but were not confirmed by any means, and should not be relied upon. Quantification of peaks was based on a mixed standard of several organic compounds.

DISCUSSION

Variability in sample components

The results for both specific assays and for general properties reveal that except in a very broad sense, there is essentially no correlation between constituents of greywater permeates. In general, each component tested varies independently of other properties. The main general measure of pollution, COD, does not vary proportionally with protein, carbohydrate, surfactant, extractable organic compounds, nor with measures of total material such as TDS and NVM.

Variation between samples in concentration of particular components also varies. Most assays yielded results that varied over a full order of magnitude, or more.

Two main differences were apparent between permeates derived from Naval Academy waste and that derived from shipboard waste. The shipboard samples were uniformly acidic while the academy samples were alkaline. The pH of the waste may be affected by many different factors. Soaps and laundry detergents are usually basic while citrus beverages and vinegar are acidic. The difference in pH is no doubt due to differences in the cleaning agents used, the housekeeping routines, and the method of disposal of foodstuff between the Naval Academy and the ship. The second main difference was that the shipboard samples were usually (but not always) more dirty than the academy samples. This is most likely due to reduced usage of clean water aboard ship. Shipboard routines usually seek to make the most efficient use of the limited fresh water supply and the limited storage space for liquid waste. These routines must be strict enough to allow the ship to operate in certain environmentally sensitive areas where overboard disposal is not permitted(4). Free from these restrictions, waste from the Naval Academy is no doubt more dilute than that produced about ship.

Effectiveness of filtration

The COD found in all the permeate samples tested was higher than the initial goal of 5 mg/l BOD. The lowest sample tested had a COD of 122 mg/l and the highest was 1160 mg/l. Nevertheless, the filtration procedure did remove a substantial amount of the COD present in the waste feed samples (initial feed COD ranging from 418 mg/l to >2700mg/l). Approximately 60% of COD was removed by filtration.

Total Suspended Solids was also greatly reduced by filtration. Suspended particles were

essentially completely removed during the procedure. The small amount that is detected in some permeate samples is probably due to aggregation of sample molecules or bacterial growth after the sample was collected. Permeate samples were uniformly clear to the eye, but did exhibit clouding and bacterial growth upon storage.

The results of filtration on TDS and NVM is less clear. Sample 7 shows that TDS was higher in the permeate than in the feed. Ordinarily, this should not be possible. However, this may be explained if the previous day the feed was contained considerably more salt, and was perhaps even contaminated with seawater. It is possible that the filter retained a good portion of salt water from the previous day, and that this water was represented in the sample collected on the day of the analysis. The high degree of variability in the samples reported above supports this notion, however, the possibility of contamination of samples calls into question the validity of comparison between any pair of feed and permeate samples.

Comments on techniques

The actual nature of the more or less heavily contaminated waste water studied in this report is the main weakness of the experiments. Most chemical or biochemical assays are designed to work most accurately on somewhat pure, dilute samples. Since these waste samples are not pure, not dilute, and contaminated with unknown compounds, interferences with the assays are likely to be significant and unknown.

The assay least likely to be affected is NVM, which employs a simple evaporation of the water, even though this assay will loose any compounds that boil below or near 100°C. However, he results of the GC/MS experiments show that the total organic compounds of sufficient volatility is very small, perhaps no more than a few milligrams per liter.

Measurement of TDS also is fairly reliable. The one caution that needs to be acknowledged is that uncharged compounds, such as carbohydrates and polysaccharides, will not be detected due to the lack of contribution to conductivity.

Other assays, especially, those dealing with protein and carbohydrate are subject to various interferences. As the bulk of material in the waste water samples is unidentified, it is impossible to predict whether the interferences will yield falsely high or falsely low readings, or if the effect will be significant. The protein assay actually determines the concentration of certain amino acids, and will give false high readings if those amino acids are present in the solution(5).

The main weakness of the molecular weight determination is the limitation of the technique to UV absorbing compounds. The determination of COD in these filtered samples was not possible due to interference from glycerin present on the filter membrane as a preservative. Even after 4 successive washes, the contribution to COD by the remaining glycerin was too large to allow a viable assay. While many different organic compounds absorb light in the UV range, there is no guarantee that the overall UV absorbance is proportional to organic content. Also, it is probable that certain highly absorbent compounds are present and dominate the UV absorbance signal. The results of the filtration of waste samples may reflect the filtration of relatively few compounds rather than the total bulk organic content.

The following comments are derived from the Orbeco "Quick'n'Easy" Test Outfit instruction and documentation manual(6). Potential interferences or problems are listed for each specific assay.

Chloride: bromide, iodide, cyanide, sulfite, thiosulfate, and sulfide.

Chlorine: chromate, manganese, bromine, iodine, ozone, chloroisocyanurate, chlorine

dioxide, hydrogen peroxide. Chlorine concentration is not stable in samples.

Copper: Organic bound copper may be unreactive.

Lead: bismuth, tin, thallium, silver, copper, mercury, chlorine. Assay may be sensitive to trace metal in labware.

Nitrate: nitrite.

Sulfide: thiosulfide, iodide, strong reducing agents.

Surfactant: Test sometimes limited by failure of waste sample to separate in chloroform extraction required by the kit.

REFERENCES

1) DoD Directive 5090.1B, (1996) Chapter 19, Environmental compliance afloat.

2) Eaton, A. D., Clesceri, L. S., and Greenberg, A. E., *Standard Methods for the Examination of Water and Wastewater*, 19th ed. (American Public Health Association, Washington, D.C., 1995), Method 5210 and 5220.

3) Viles, F.J. Jr., and Silverman, L. (1949) Determination of starch and cellulose with anthrone. Anal Chem 21:950-953.

4) DoD Directive 5090.1B, (1996) Chapter 19, Environmental compliance afloat. Section19-3.4.1.

5) Ohnishi, S. T. And Barr J. K. (1978) A simplified method of quantitating proteins using the biuret and phenol reagents. Anal Biochem 86:193

6) ----------, *Operating Instructions, Series 952 Aqua Analyser 2.* (Orbeco-Hellige, Farmingdale, New York, 1987).

Table 1.

Sample	Date	Origin	Source
1	2 May 1996	Annapolis	greywater and sewage
2	22 May 1996	Annapolis	greywater
3	29 May 1996	Annapolis	greywater
4	4 June 1996	Annapolis	greywater
5	19 June 1996	Annapolis	greywater
7	18 July 1996	DD997	greywater and sewage
8	18 July 1996	DD997	Feed for sample 7
9	28 July 1996	DD997	greywater and sewage
10	28 July 1996	DD997	Feed for sample 9
11	21 Sept. 1996	CG51	greywater and sewage
12	21 Sept. 1996	CG51	Feed for sample 11
13	25 Sept. 1996	CG51	greywater and sewage
14	25 Sept 1996	CG51	Feed for sample 12

Table 2. Constituents of Permeates

Assay	1	2	3	4	5	7	9	11	13	Avg
pH	9.3	9.4	9.4	10.0	8.1	5.9	4.9	6.8	6.4	7.8
TDS	1232	989	744	978	1071	5480	546	5560	8140	2749
TSS	<1	<1	<1	<1	6	<1	<1	13	<1	-
NVM	1780	1270	877	1576	860	2724	218	5866	7799	2552
COD	800	166	510	300	122	160	1160	385	612	468
Extractable	18	18	70	14	16	-	-	-	-	27
Protein	45	50	61	40	160	20	38	100	-	64
Carbohydrate	-	-	-	-	14	4	5	6	-	7
Phosphate	>5.3	183	73	61	94	11	31	-	-	76
Nitrate	-	-	0.31	0.18	0.05	0.02	0.08	-	-	0.13
Surfactant	>17	>33	17	42	11	3	20	-	-	19
Chlorine	0.26	0.08	0.64	0.29	0.02	0.09	-	-	-	0.23
Chloride	36	8.4	>50	>50	26	>50	19	-	-	22
Copper	<0.02	0.08	0.15	0.04	0.07	0.32	1.6	<0.02	-	0.45
Lead	-	-	-	-	0.08	0.03	<.01	-	-	0.06
Zinc	-	-	0.06	0.24	0.34	0.07	0.23	<0.02	-	0.19
Sulfide	-	-	0.15	0.05	0.52	0.02	0.01	0.02	-	0.13
Hardness	-	-	1.4	11	15	>30	13	-	-	10

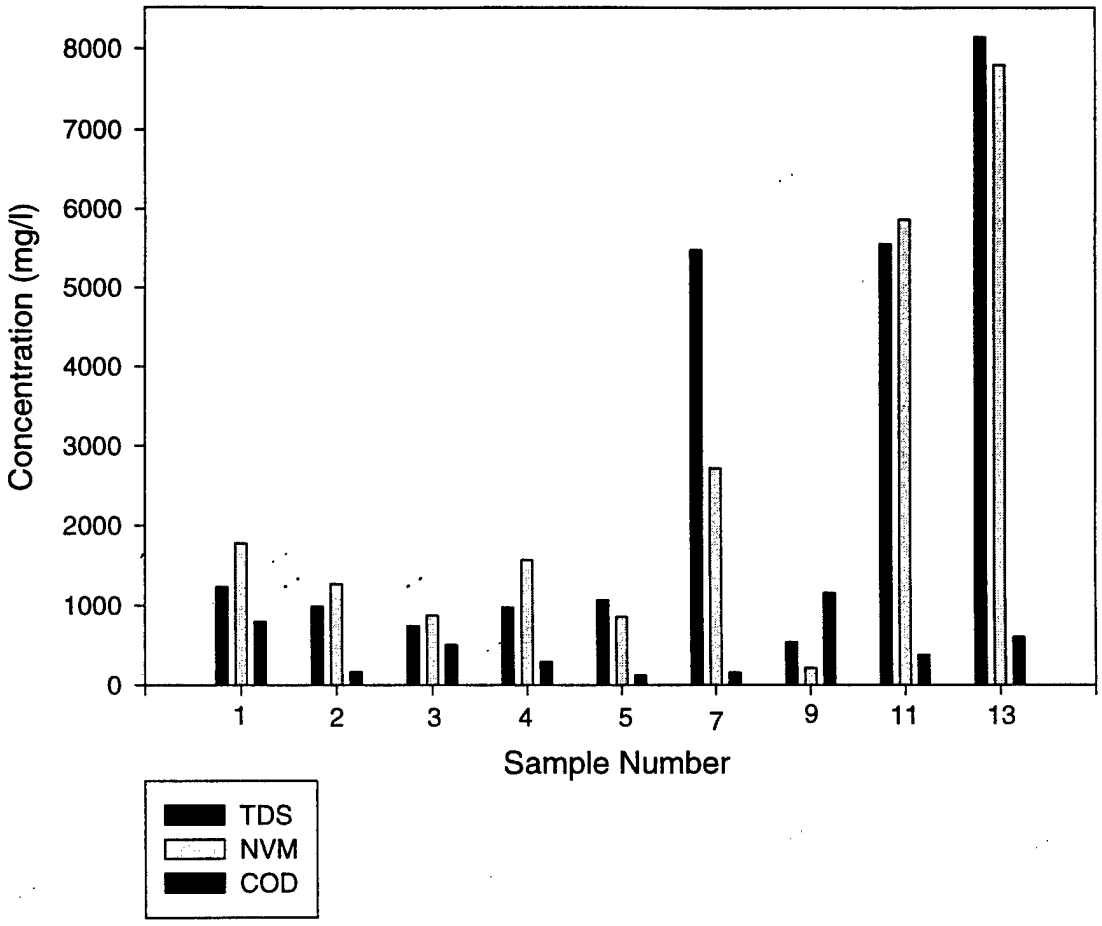

Figure 1. Gross constituents of permeates. Permeate samples recovered after filtration treatment of greywater or greywater/sewage were tested for total material in the form of Total Dissolved Solids, Non-Volatile Material, and Chemical Oxygen Demand.

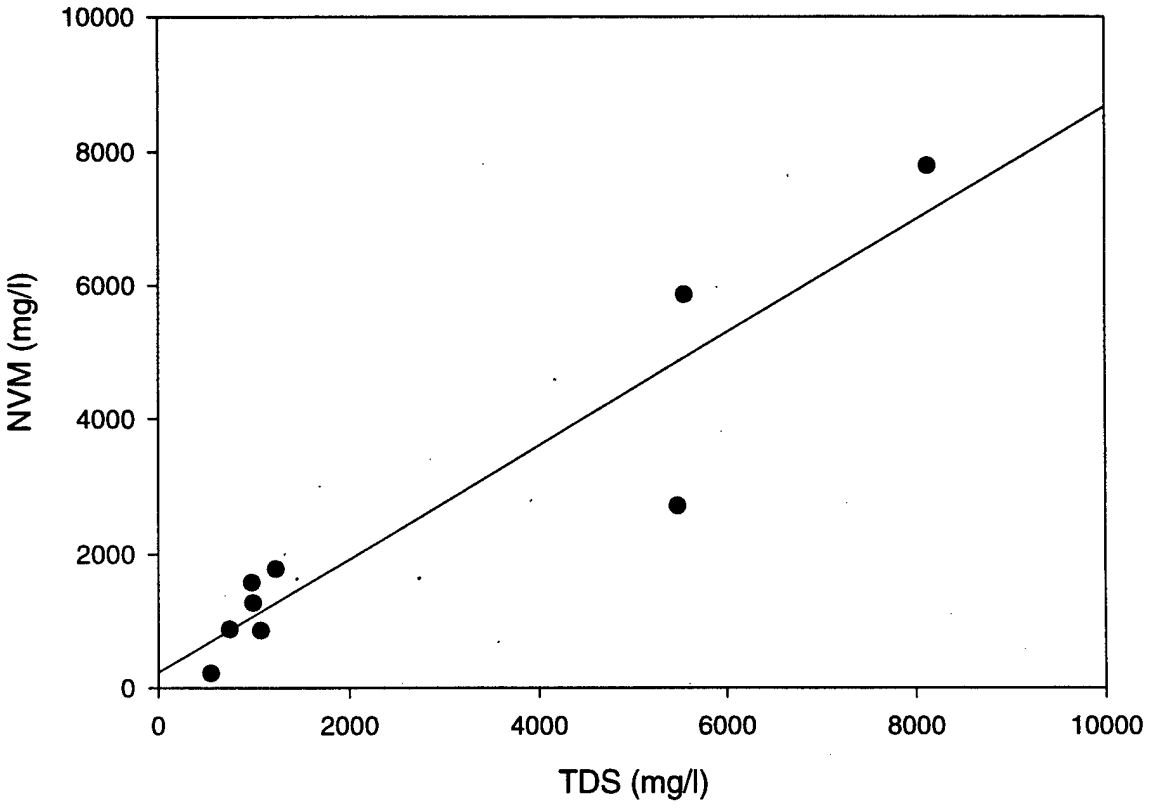

Figure 2. Sample components do not correlate. For each sample, Total Dissolved Solids was plotted against Non-Volatile Material(A). NVM(o) and TDS(•) were plotted against Chemical Oxygen Demand(B). TDS(•) and COD(o) were plotted against protein concentration(C).

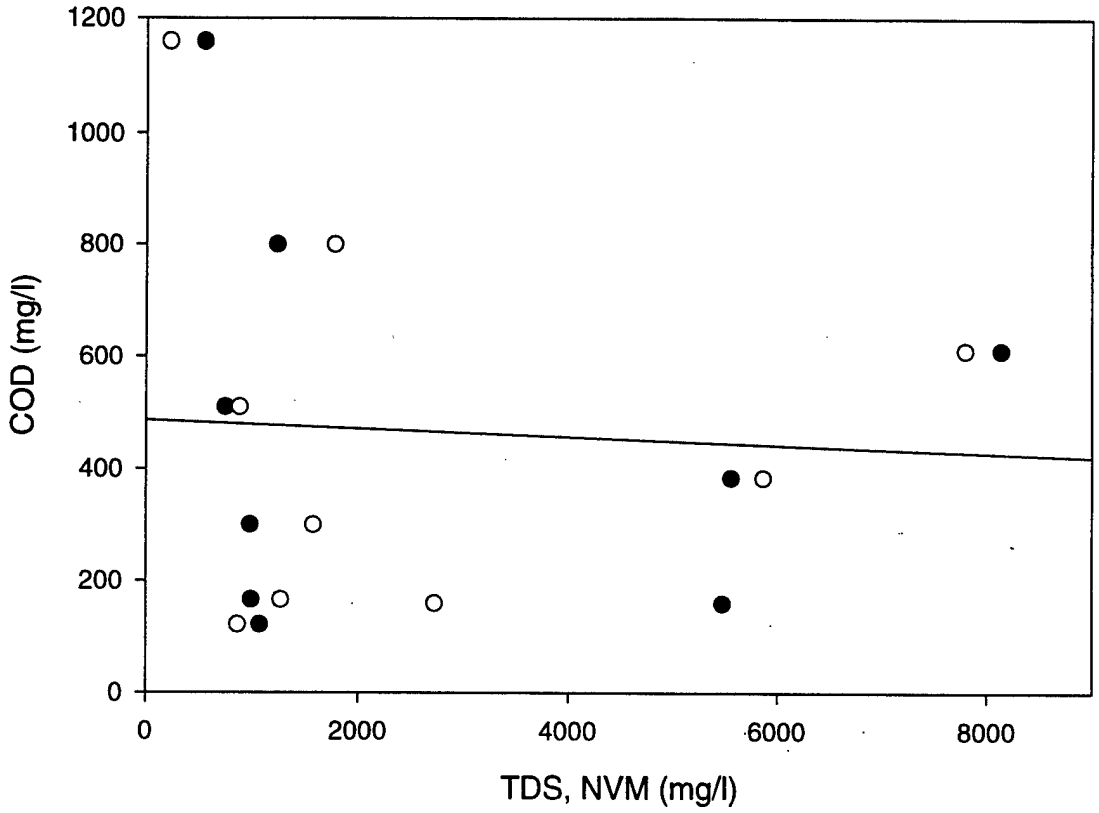

Figure 2. (Continued) Sample components do not correlate. For each sample, Total Dissolved Solids was plotted against Non-Volatile Material(A). NVM(o) and TDS(•) were plotted against Chemical Oxygen Demand(B). TDS(•) and COD(o) were plotted against protein concentration(C).

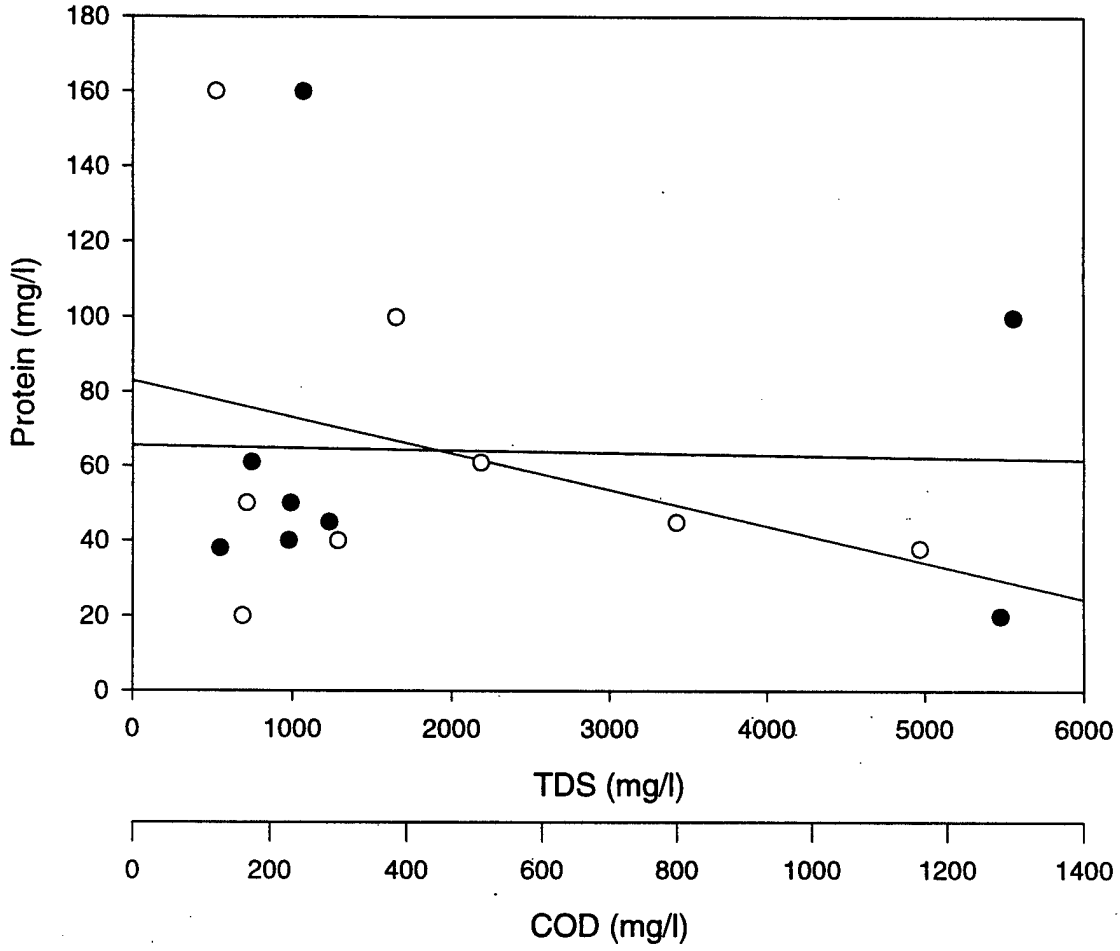

Figure 2. (Continued) Sample components do not correlate. For each sample, Total Dissolved Solids was plotted against Non-Volatile Material(A). NVM(o) and TDS(•) were plotted against Chemical Oxygen Demand(B). TDS (•) and COD(o) were plotted against protein concentration(C).

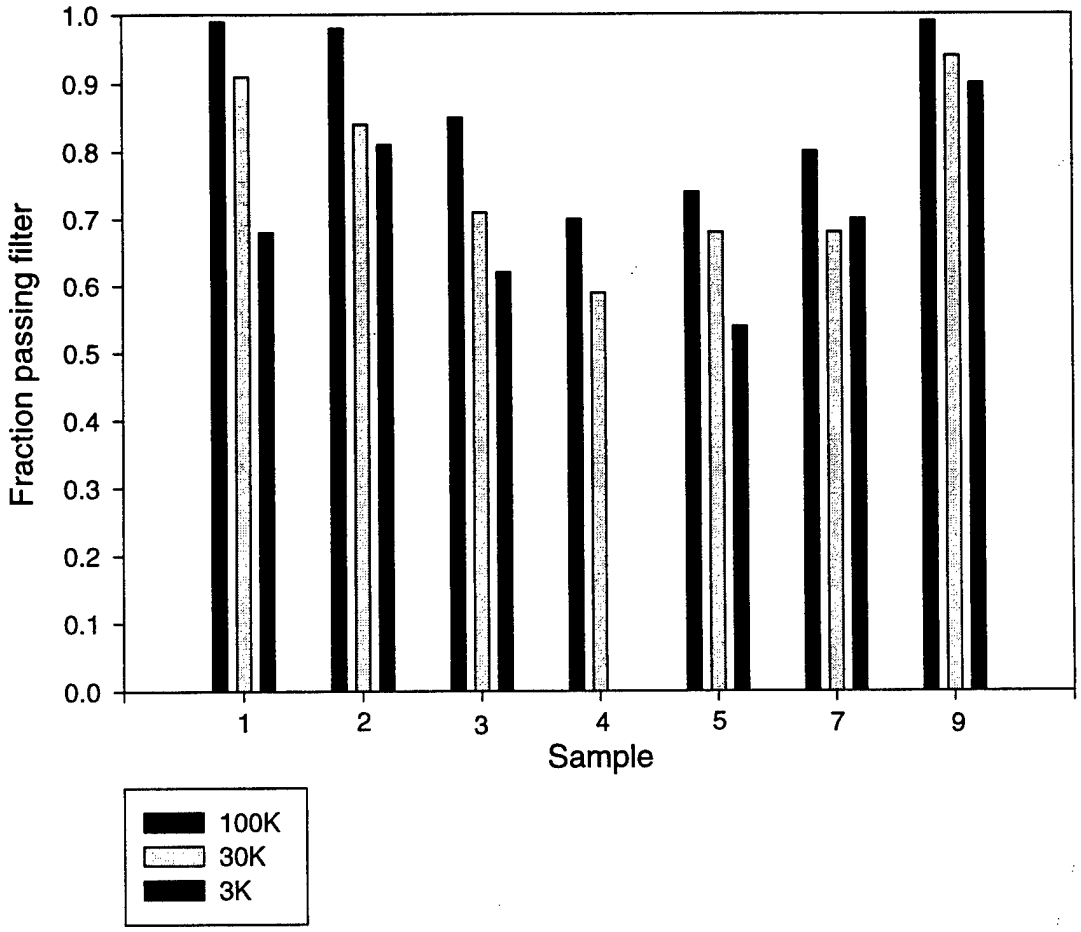

Figure 3. The bulk of UV-absorbing material is of low molecular weight. Permeate samples were subjected to centrifuge ultrafiltration with a molecular weight cutoff of 100,000 Daltons, 30,000 Daltons, and 3,000 Daltons. The passage of organic molecules was measured by absorbance and 205nm. The fraction of the original starting absorbance passing the filter reveals that amount of material below the molecular weight cutoff.

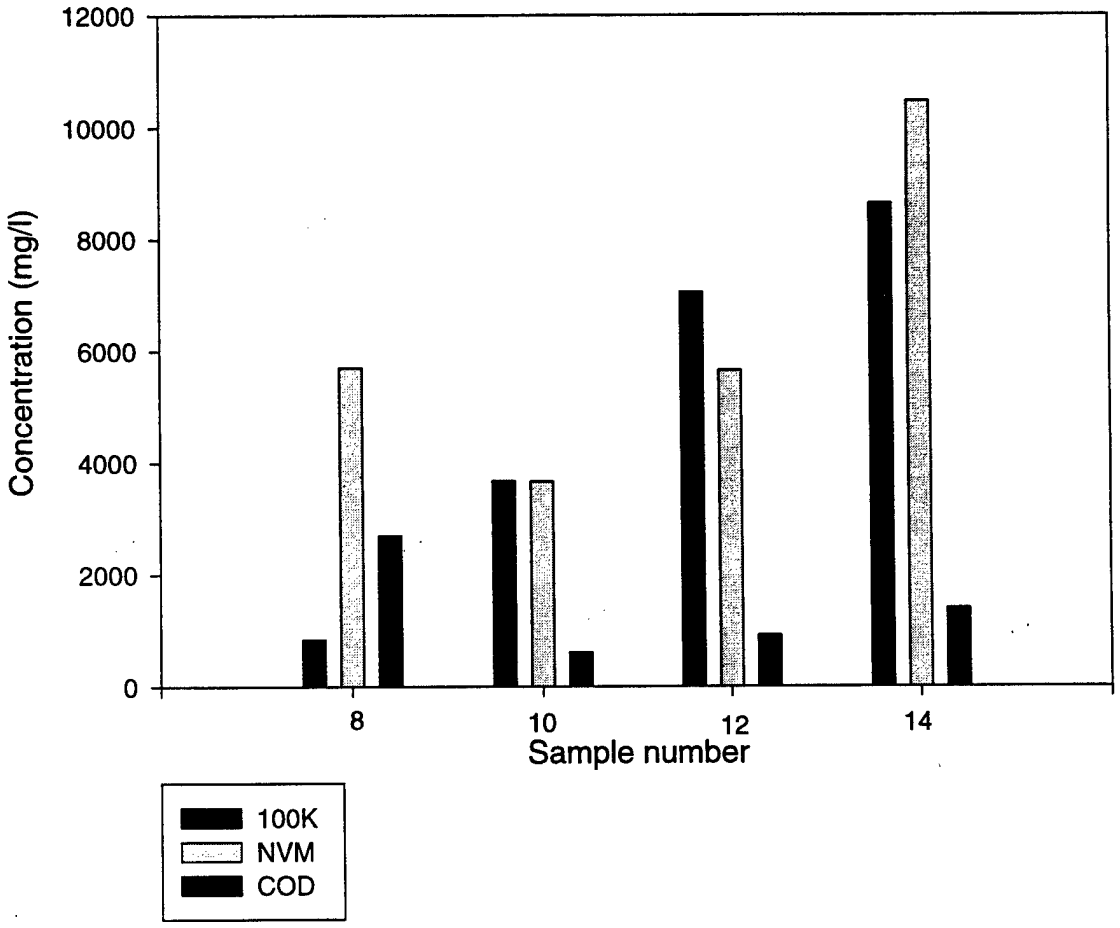

Figure 4. Gross constituents of shipboard non-oily wastewater feed. Feed samples were collected and tested for TDS, NVM, and COD as in figure 1.

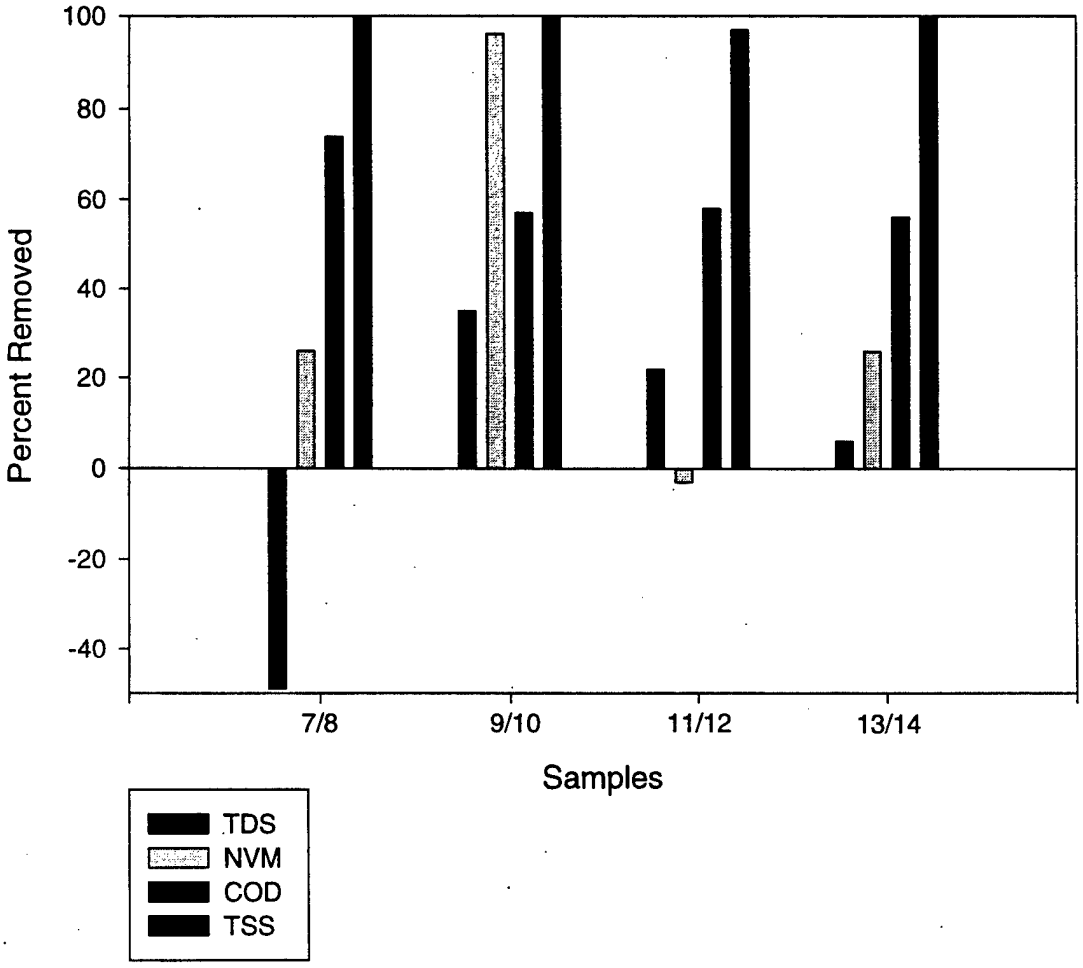

Figure 5. Removal of gross constituents by the filtration treatment. Sample feed/permeate pairs were compared as indicated. Percent Removed was arrived at by dividing the permeate value by the feed value and multiplying by 100, then subtracting that number from 100%.

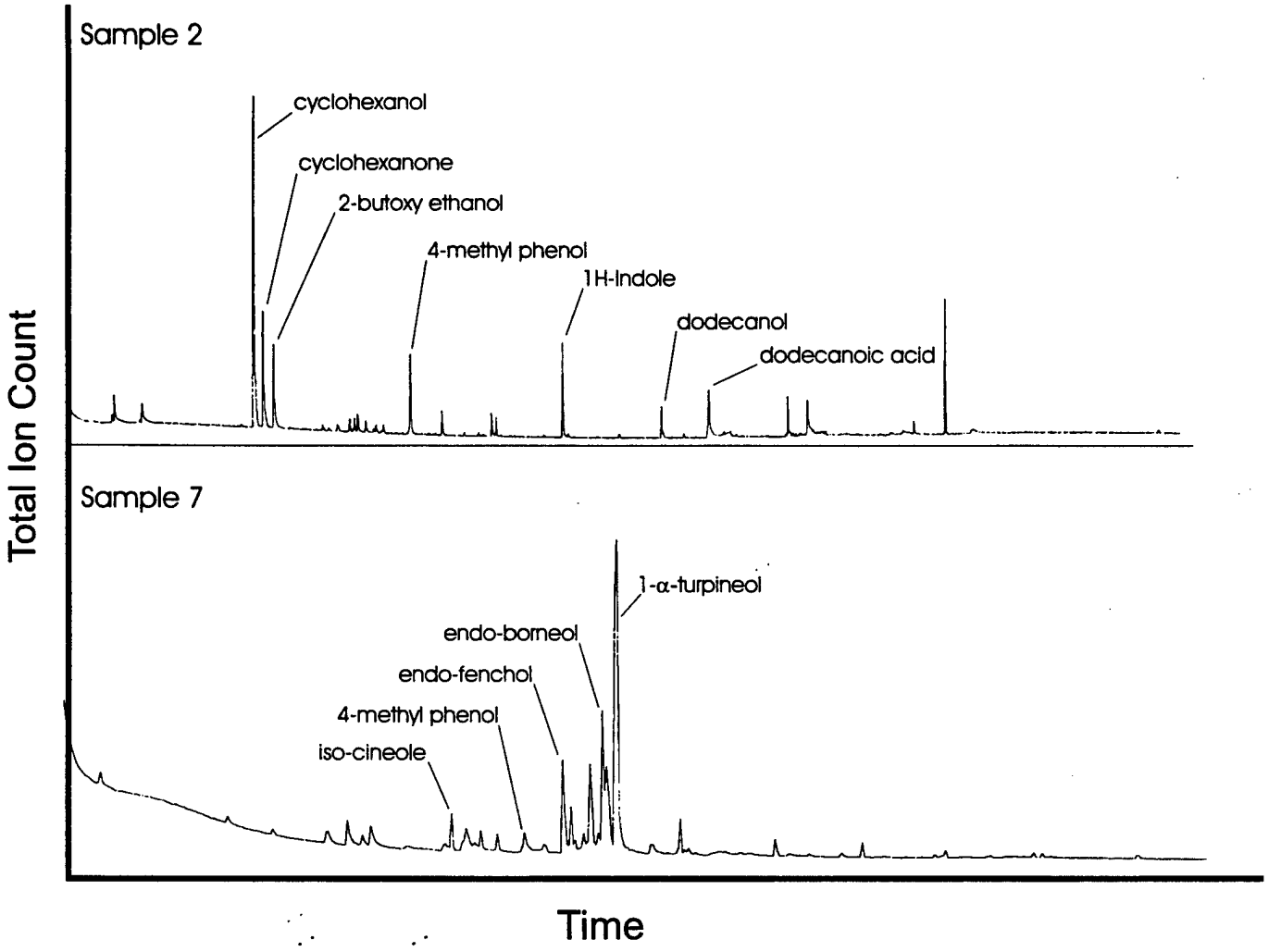

Figure 6. Gas chromatography of volatile compounds. Samples were injected into a Model 5890 Gas Chromatograph, at an initial temperature of 40°C. The temperature was raised to 250°C over 20 minutes. Total ion count is shown, with compond identities.

APPENDIX

The following pages contain the individual reports prepared for each sample.

Center for Bio/Molecular Science and Engineering
U.S. Naval Research Laboratory

4555 Overlook Avenue
Washington, DC 20375

Water Analysis

Sample number: 1

Date: 5/10/96
Prepared by: Dan Zabetakis
Phone: (202) 404-6071
E-mail: dan@cbmse.nrl.navy.mil

Sample Information:

Test Equipment	LP-2
Membrane	Zenon MF-100
Wastewater	Greywater and sewage
Time	2 May 1996 at 0700 hours
Concentration	7:1
Operating Conditions	20:1 (greywater to sewage) feed and bleed
Transmembrane Pressure	40

Physical Appearance:

Light yellow clear solution. Odor strong but not foul. Solution foamed easily upon shaking or aspiration. No particulates or microorganisms detected. A Portion of the sample stored at room temperature for 1 week showed vigorous bacterial growth.

Characteristics:

Temperature	23°C		
pH	9.3	Total dissolved solids	1232 mg/l
Conductivity	1819 µmho/cm	Resistivity	<0.001 MΩ/cm

Constituents:

Non-Volatile Material	1780 mg/l
Extractable Non-Volatile Material	18 mg/l

Protein	45 mg/l
Chloride	36 mg/l
Chlorine, free	0.21 mg/l
Chlorine, total	0.26 mg/l
Phosphate, total	>5.3 mg/l
Copper	<0.02 mg/l
Surfactant	>16.6 mg/l

UV Absorbance and Filtration:

UV Spectrum	Single peak at 280 nm (OD 0.5250)	
1.0 µm filterable particles	<1.0 mg/L	
filter	OD at 280 nm	relative abundance
0.22 µm	0.5192	1
100 kD cutoff	0.5142	0.99
30 kD cutoff	0.4721	0.91
3 kD cutoff	0.3528	0.68

COD and Gel Filtration Chromatography:

COD	800 mg/l	
MW fraction	% COD	% COD for KHP
VL	7.5	8.0
VL - 10^6	0	8.0
10^6 - 4000	31.3	39.8
4000 - 50	35.8	44.3
50 - 0	25.4	0
Void	0	0
KHP= Potassium hydrogen phthalate (MW=204)		
VL= very large Void= Fraction collected after theoretical zero MW		

Gas Chromatography, Mass Spectrometry:

Compound	Amount
Cyclohexanol	0.15 mg/l
4-methyl phenol (?)	0.05 mg/l
1H-Indole	0.24 mg/l
Dodecanoic acid	0.05 mg/l
Number of minor peaks	36

X-Ray Fluorimetry:

No significant metals detected.

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Water Analysis

Sample Number: 2

Date: 5/21/96
Prepared by: Dan Zabetakis
Phone: (202) 404-6071
E-mail: dan@cbmse.nrl.navy.mil

Sample Information:

Test Equipment	LP-5
Membrane	MF-100, membrane 8
Wastewater	Greywater
Time	22 May 1996 at 0730 hours
Operating Conditions	50:1 feed and bleed
Transmembrane Pressure	45 psi

Physical Appearance:

Light yellow-green clear solution. Moderate foaming upon shaking. Odor moderate. No microorganism detected.

Characteristics:

Temperature	24.4°C		
pH	9.4	Total dissolved solids	989 mg/l
Conductivity	1485 µmho/cm	Resistivity	<0.001 MΩ/cm

Constituents:

Non-Volatile Material	1270 mg/l
Extractable Non-Volatile Material	18 mg/l
Protein	50 mg/l
Chloride	8.4 mg/l
Chlorine, free	0.04 mg/l

Chlorine, total	0.075 mg/l
Phosphate, total	183 mg/l
Copper	0.08 mg/l
Surfactant	>33.2 mg/l

UV Absorbance and Filtration:

UV Spectrum peaks	OD 280nm	0.453	OD 205nm	2.979
1.0µm filterable particles	<1.0 mg/l			

filter	OD_{280}	rel. abund.	OD_{205}	rel. abund.
0.22 µm	0.4258	1	2.881	1
100 kD cutoff	0.4190	0.98	2.795	0.97
30 kD cutoff	0.3596	0.84	2.639	0.91
3 kD cutoff	0.3452	0.81	2.360	0.82

COD and Gel Filtration Chromatography:

COD	166 mg/l
The COD value was too low to allow determination of fractions by GFC.	

Gas Chromatography. Mass Spectroscopy:

Compund	Amount
unknown	0.05 mg/l
cyclohexanol	0.62 mg/l
cyclohexanone	0.20 mg/l
2-butoxy ethanol (?)	0.15 mg/l
4-methyl phenol	0.12 mg/l
benzeneethanol	0.04 mg/l
unknown	0.03 mg/l
1H-indole	0.11 mg/l
1-dococanol	0.04 mg/l
dodecanoic acid	0.11 mg/l

unknown	0.05 mg/l
tetradecanoic acid	0.09 mg/l
unknown	0.13 mg/l
Number of minor peaks:	37

X-ray Flourimetry:

No significant metals detected.

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Washington, DC 20375

Water Analysis

Sample Number: 3

Date: 3 June 1996
Prepared by: Dan Zabetakis
Phone: (202) 404-6071
E-mail: dan@cbmse.nrl.navy.mil

Sample Information:

Test Equipment	LP-5
Membrane	MF-100, Membrane 8
Time	29 May 1996 at 1030 hours

Physical Appearance:

Light yellow solution, very easily foams. Odor mild.

Characteristics:

Temperature	22.8°C		
pH	9.4	Total dissolved solids	744 mg/l
Conductivity	1110 µS/cm	Resistivity	<.001 MΩ/cm

Constituents:

Non-Volatile Material	877 mg/l
Extractable Non-Volatile Material	70 mg/l
Protein	61 mg/l
Chloride	>50 mg/l
Chlorine, free	0.39 mg/l
Chlorine, total	0.64 mg/l
Phosphate, total	73 mg/l
Copper	0.15 mg/l
Surfactant	17 mg/l

Zinc	0.06 mg/l
Sulfide	0.15 mg/l
Hardness (as $CaCO_3$)	1.4 mg/l
Nitrate	0.31 mg/l

UV Absorbance and Filtration:

UV Spectrum peaks	OD 280nm	0.3712	OD 205nm	2.955
1.0µm filterable particles	<1.0 mg/l			
filter	OD_{280}	rel. abund.	OD_{205}	rel. abund.
100 kD cutoff	0.3146	0.85	2.922	0.99
30 kD cutoff	0.2635	0.71	2.835	0.96
3 kD cutoff	0.2311	0.62	2.699	0.91

COD and Gel Filtration Chromatography:

COD	510 mg/l
Fraction	Percent COD (VL= very large; Void= below zero MW)
VL	34.8
VL - 10^6	0
10^6 - 4000	47.8
4000 - 50	17.4
50 - 0	0
Void	0

Gas Chromatography. Mass Spectroscopy:

Compound	Amount
unknown	0.04 mg/l
cyclohexanol	0.15 mg/l
cyclohexanone	0.08 mg/l
4-methyl phenol	0.19 mg/l
3-methyl indole	0.03 mg/l

dodecanoic acid	0.04 mg/l
tetradecanoic acid	1.39 mg/l
hexadecanoic acid	1.25 mg/l
octadecanoic acid	2.34 mg/l
Number of minor peaks:	36

X-ray Fluorimetry:

No significant metals detected.

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4555 Overlook Avenue
Washington, D.C. 20375

Water Analysis

Sample Number: 4

Date: 7 June 1996
Prepared by: Dan Zabetakis
Phone: (202) 404-6071
E-mail: dan@cbmse.nrl.navy.mil
Technical Assistance: Laura Wells

Sample Information:

Test Equipment	LP-5
Membrane	Zenon MF-100
Wastewater	Greywater
Time	1050 hours 4 June 1996
Operating Conditions	Feed and Bleed
Transmembrane Pressure	40 psi (?)

Physical Appearance:

Light yellow clear solution. Odor mild and soapy.

Characteristics:

Temperature	30.6°C		
pH	10.0	Total dissolved solids	978 mg/l
Conductivity	1518 µmho/cm	Resistivity	<0.001 MΩ/cm

Constituents:

Non-Volatile Material	1576 mg/l
Extractable Non-Volatile Material	14 mg/l
Protein	40 mg/l
Chloride	>50 mg/l
Chlorine, free	0.08 mg/l

Chlorine, total	0.29 mg/l
Phosphate, total	60.5 mg/l
Copper	0.035 mg/l
Zinc	0.24 mg/l
Hardness (as $CaCO_3$)	10.9 mg/l
Nitrate	0.18 mg/l
Sulfide	0.054 mg/l
Surfactant	41.5 mg/l

UV Absorbance and Filtration:

UV Spectrum peaks	OD 280nm	0.4486	OD 205nm	3.1279
1.0µm filterable particles	<1.0 mg/l			
filter	OD_{280}	rel. abund.	OD_{205}	rel. abund.
100 kD cutoff	0.3123	0.70	2.9220	0.93
30 kD cutoff	0.2649	0.59	2.8244	0.9
3 kD cutoff	N/A	N/A	2.5514	0.82

COD and Gel Filtration Chromatography:

COD	300 mg/l
Due to low COD value, GFC determination was not possible.	

Gas Chromatography. Mass Spectroscopy:

Compound	Amount
cyclohexanol	0.13 mg/l
cyclohexanone	0.09 mg/l
unknown	0.01 mg/l
4-methyl phenol	0.04 mg/l
unknown	0.01 mg/l
indole	<0.01 mg/l
unknown	0.02 mg/l
Number of minor peaks:	19

X-ray Fluorimetry:

No significant metals detected.

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Water Analysis

Sample Number:

Date: 21 June 1996
Prepared by: Dan Zabetakis
Phone: (202) 404-6071
E-mail: dan@cbmse.nrl.navy.mil
Technical Assistance: Laura Wells

Sample Information:

Test Equipment	LP-5
Membrane	Zenon MF-100
Wastewater	Greywater
Concentration	50:1
Time	1040 hours, 19 June 1996
Operating Conditions	feed and bleed
Transmembrane Pressure	40 psi

Physical Appearance:

Light yellow clear solution. Mild musty odor. Rod-shaped bacteria observed with heavy bacterial growth after 2 days at room temperature.

Characteristics:

Temperature	26.5°C		
pH	8.1	Total dissolved solids	1071 mg/l
Conductivity	1679 µmho/cm	Resistivity	<0.001 MΩ/cm

Constituents:

Non-Volatile Material	860 mg/l
Extractable Non-Volatile Material	16 mg/l
Acid Extractable NVM	24 mg/l

Carbohydrate (as glucose)	13.9 mg/l
Protein	160 mg/l
Chloride	26.2 mg/l
Chlorine, free	0.02 mg/l
Chlorine, total	0.02 mg/l
Phosphate, total	93.5 mg/l
Copper	0.07 mg/l
Surfactant (as LAS)	11 mg/l
Lead	0.078 mg/l
Nitrate (as N)	0.05 mg/l
Hardness, total (as $CaCO_3$)	15.0 mg/l
Sulfide	0.52 mg/l
Zinc	0.34 mg/l

UV Absorbance and Filtration:

UV Spectrum peaks	OD 280nm	0.4237	OD 205nm	2.7176
1.0µm filterable particles	6 mg/l			

filter	OD_{280}	rel. abund.	OD_{205}	rel. abund.
100 kD cutoff	0.3116	0.735	2.5190	0.927
30 kD cutoff	0.2859	0.675	2.3719	0.873
3 kD cutoff	0.2267	0.535	2.1333	0.785

COD and Gel Filtration Chromatography:

COD	122 mg/l
COD was too low to determine MW fractions.	

Gas Chromatography. Mass Spectroscopy:

Compound	Amount
cyclohexanol	0.27 mg/l
unknown	0.05 mg/l

unknown	0.03 mg/l
phenol (?)	0.03 mg/l
4-methyl phenol	0.04 mg/l
indole	0.06 mg/l
Number of minor peaks:	21

X-ray Flourimetry:

No significant metals detected.

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4555 Overlook Avenue
Washington, DC 20375

Water Analysis

Sample Number: 7

Date: 31 July, 1996
Prepared by: Dan Zabetakis
Phone: (202) 404-6071
E-mail: dan@cbmse.nrl.navy.mil
Technical Assistance: Laura Wells

Sample Information:

Test Equipment	
Membrane	
Wastewater	Non-Oily Permeate
Time	18 July 1996 at 22:00 hours
Operating Conditions	
Transmembrane Pressure	

Physical Appearance:

Clear light yellow solution. Musty odor. No bacteria observed.

Characteristics:

Temperature	21.4 °C		
pH	5.9	Total dissolved solids	5480 mg/l
Conductivity	8.09 mS/cm	Resistivity	--

Constituents:

Non-Volatile Material	2724 mg/l
Extractable Non-Volatile Material	
Carbohydrate (as glucose)	4 mg/l
Protein	20 mg/l
Chloride	>46 mg/l

Chlorine, free	0.02 mg/l
Chlorine, total	0.09 mg/l
Phosphate, total	11.1 mg/l
Copper	0.315 mg/l
Surfactant	3.25 mg/l
Lead	0.028 mg/l
Nitrate	0.02 mg/l
Hardness, total (as $CaCO_3$)	>30 mg/l
Sulfide	0.017 mg/l
Zinc	0.07 mg/l

UV Absorbance and Filtration:

UV Spectrum peaks	OD 280nm	0.1784	OD 205nm	0.8088
1.0µm filterable particles	<1.0 mg/l			
filter	OD_{280}	rel. abund.	OD_{205}	rel. abund.
1.0 µm	0.1302	73.6	0.5528	61.3
100 kD cutoff	0.1415	79.8	0.7062	84.5
30 kD cutoff	0.1205	68.3	0.5765	64.8
3 kD cutoff	0.1233	69.8	0.5962	67.8

COD and Gel Filtration Chromatography:

COD	160 mg/l

Gas Chromatography. Mass Spectroscopy:

Compound	Amount
Unknown A	0.15 mg/l
Unknown B	0.23 mg/l
Unknown	0.25 mg/l
Unknown	0.21 mg/l

Unknown C	0.40 mg/l
Isocineol	0.19 mg/l
Unknown	0.14 mg/l
4-methyl phenol	0.25 mg/l
Endo-fenchol	1.05 mg/l
Unknown	0.98 mg/l
Endo-Borneol	1.90 mg/l
1-terpineol	3.31 mg/l
Unknown D	0.18 mg/l
Unknown	0.09 mg/l
Number of minor peaks:	23

X-ray Flourimetry:

No significant metals detected.

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4555 Overlook Avenue
Washington DC 20375

Water Analysis

Sample Number: 8

Date: 31 July 1996
Prepared by: Dan Zabetakis
Phone: (202) 404-6071
E-mail: dan@cbmse.nrl.navy.mil
Technical Assistance: Laura Wells

Sample Information:

Test Equipment	
Membrane	
Wastewater	Non-Oily Feed
Time	18 July 1996 at 2200 hours
Operating Conditions	
Transmembrane Pressure	

Physical Appearance:

Brown solution. Suspended particles evident. Odor strong and musty. Bacteria observed.

Characteristics:

Temperature	21.2 °C		
pH	5.8	Total dissolved solids	3690 mg/l
Conductivity	5.64 mS/cm	Resistivity	--

Constituents:

Non-Volatile Material	3679 mg/l
Extractable Non-Volatile Material	
Carbohydrate (as glucose)	3 mg/l
Protein	120 mg/l
Chloride	33 mg/l

Chlorine, free	1.25 mg/l
Chlorine, total	1.10 mg/l
Phosphate, total	30.5 mg/l
Copper	1.03 mg/l
Surfactant	5.0 mg/l
Lead	0.053 mg/l
Nitrate	<0.005 mg/l
Hardness, total (as $CaCO_3$)	>30 mg/l
Sulfide	0.33 mg/l
Zinc	1.33 mg/l

UV Absorbance and Filtration:

UV Spectrum peaks	OD 280nm	1.2544	OD 205nm	2.1227
1.0µm filterable particles	1070 mg/l			
filter	OD_{280}	rel. abund.	OD_{205}	rel. abund.
1.0 µm	0.2484	20.1	0.5912	22.4
100 kD cutoff	0.1985	16.1	0.4619	15.9
30 kD cutoff	0.1187	9.8	0.2585	5.6
3 kD cutoff	0.1130	9.3	0.2722	6.3

COD and Gel Filtration Chromatography:

COD	610 mg/l

Gas Chromatography. Mass Spectroscopy:

Compound	Amount
3-hydroxy-2-butanone	2.33 mg/l
Unknown A	0.19 mg/l
Unknown B	0.36 mg/l
Unknown	0.17 mg/l

Unknown	0.37 mg/l
Unknown	0.28 mg/l
Unknown	0.24 mg/l
Unknown C	0.90 mg/l
Unknown	0.22 mg/l
Endo-fenchol	1.23 mg/l
Unknown	1.62 mg/l
Endo-borneol	2.53 mg/l
1-terpineol	4.40 mg/l
Benzene derivative	0.30 mg/l
Unknown D	0.19 mg/l
Unknown	0.17 mg/l
Decanoic Acid	1.20 mg/l
Dodecanoic Acid	0.77 mg/l
Tetradecanoic acid	0.76 mg/l
Octadecane	0.07 mg/l
Hexanoic Acid	1.13 mg/l
Number of minor peaks:	31

X-ray Flourimetry:

No significant metals detected.

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Water Analysis Comparison

Sample Numbers: 7 and 8

Date: 1 August 1996
Prepared by: Dan Zabetakis
Phone: (202) 404-6071
E-mail: dan@cbmse.nrl.navy.mil
Technical Assistance: Laura Wells

Parameter:	Percent remaining in permeate:
Non-Volatile Material	74%
COD	26%
Total Dissolved Solids	149%
UV Absorbance (205nm)	38%
UV Absorbance (280nm)	14%
Total Suspended Solids	<0.09%
Carbohydrate	133%
Protein	17%
Chloride	139%
Chlorine, free	1.6%
Chlorine, total	8.2%
Phosphate	36%
Copper	31%
Surfactant	65%
Lead	53%
Nitrate	>400%
Sulfide	5.2%
Zinc	5.3%
Unknown A	79%
Unknown B	64%
Unknown C	44%
Endo-fenchol	85%
Endo-Borneol	76%
1-terpineol	75%
Unknown D	95%

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Washington, DC 20375

Water Analysis

Sample Number: 9

Date: 20 August 1996
Prepared by: Dan Zabetakis
Phone: (202) 404-6071
E-mail: dan@cbmse.nrl.navy.mil
Technical Assistance: Laura Wells

Sample Information:

Test Equipment	
Membrane	
Wastewater	Permeate
Time	30 July 1996
Operating Conditions	
Transmembrane Pressure	

Physical Appearance:

Clear yellow solution. Musty odor. No bacterial observed.

Characteristics:

Temperature	21.4°C		
pH	4.9	Total dissolved solids	546 mg/l
Conductivity	816 µS/cm	Resistivity	--

Constituents:

Non-Volatile Material	218 mg/l
Carbohydrate (as glucose)	5 mg/l
Protein	38 mg/l
Chloride	19.4 mg/l
Chlorine, free	0.56 mg/l

Chlorine, total	
Phosphate, total	30.75 mg/l
Copper	1.58 mg/l
Surfactant	20 mg/l
Lead	0.00058 mg/l
Nitrate	0.08 mg/l
Hardness, total (as $CaCO_3$)	12.8 mg/l
Sulfide	0.013 mg/l
Zinc	0.23 mg/l

1.0µm filterable particles	<1.0 mg/l

COD and Gel Filtration Chromatography:

COD	1160 mg/l

Gas Chromatography. Mass Spectroscopy:

Compound	Amount
Unknown	2.15 mg/l
1-Terpineol	2.30 mg/l
Number of minor peaks:	16

X-ray Flourimetry:

No significant metals detected.

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Washington, DC 20375

Water Analysis

Sample Number: 10

Date: 20 August 1996
Prepared by: Dan Zabetakis
Phone: (202) 404-6071
E-mail: dan@cbmse.nrl.navy.mil
Technical Assistance: Laura Wells

Sample Information:

Test Equipment	
Membrane	
Wastewater	Feed
Time	30 July 1996
Operating Conditions	
Transmembrane Pressure	

Physical Appearance:

Dark brown opaque suspension. Odor foul and strong.

Characteristics:

Temperature	21.4°C		
pH	4.2	Total dissolved solids	835 mg/l
Conductivity	1245 µS/cm	Resistivity	--

Constituents:

Non-Volatile Material	5715 mg/l
Carbohydrate (as glucose)	30 mg/l
Protein	>1000 mg/l
Chloride	
Chlorine, free	

Chlorine, total	
Phosphate, total	
Copper	
Surfactant	
Lead	
Nitrate	
Hardness, total (as $CaCO_3$)	30 mg/l
Sulfide	>1.0 mg/l
Zinc	>3.5 mg/l

1.0µm filterable particles	811 mg/l

COD and Gel Filtration Chromatography:

COD	>2700 mg/l

Gas Chromatography. Mass Spectroscopy:

Compound	Amount
1-Terpineol	4.05 mg/l
Number of minor peaks:	26

X-ray Flourimetry:

No significant metals detected.

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4555 Overlook Avenue
Washington, DC 20375

Water Analysis Comparison

Sample Numbers: 9 and 10

Date: 29 August 1996
Prepared by: Dan Zabetakis
Phone: (202) 404-6071
E-mail: dan@cbmse.nrl.navy.mil
Technical Assistance: Laura Wells

Parameter:	Percent remaining in permeate:
Non-Volatile Material	3.8%
COD	43%
Total Dissolved Solids	65%
Total Suspended Solids	<0.12%
Carbohydrate	17%
Protein	<3.8%
Hardness	42.7%
Sulfide	<1.3%
Zinc	<6.6%
1-terpineol	57%

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Water Analysis

Sample Numbers: 11(permeate), 12(feed), 13(permeate), and 14(feed).

Date: 16 October, 1996
Prepared by: Dan Zabetakis
Phone: (202) 404-6071
E-mail: dan@cbmse.nrl.navy.mil

Characteristic	Sample 11	Sample 12	Sample 13	Sample 14
Source	Permeate	Feed	Permeate	Feed
pH	6.8	6.1	6.4	5.8
Conductivity (mS/cm)	8.26	10.6	12.17	12.91
COD (mg/l)	385	920	612	1405
TDS (mg/l)	5560	7070	8140	8650
TSS (mg/l)	13	418	<0.1	445
NVM (mg/l)	5866	5673	7799	10481
Protein (mg/l)	100	175		
Carbohydrate (mg/l)	5.8	49.6		
Zinc (mg/l)	<0.02	0.28		
Sulfide (mg/l)	0.020	>4		
Copper (mg/l)	<0.02	<0.02		
OD_{205}	2.2434	2.6457		
OD_{280}	0.6357	1.3145		
100K				
205nm	1.03	0.82		
280nm	0.99	0.37		
30K				
205nm	1.01	0.78		
280nm	0.94	0.35		
3K				
205nm	0.99	0.74		
280nm	0.90	0.33		

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Washington, DC 20375

Water Analysis

Sample Numbers: 11(permeate), 12(feed), 13(permeate), and 14(feed).

Date: 16 October, 1996
Prepared by: Dan Zabetakis
Phone: (202) 404-6071
E-mail: dan@cbmse.nrl.navy.mil

Characteristic	11/12 x 100	13/14 x 100
Conductivity (mS/cm)	78%	94%
COD (mg/l)	42%	44%
TDS (mg/l)	79%	94%
TSS (mg/l)	3%	<.02%
NVM (mg/l)	103%	74%
Protein (mg/l)	57%	
Carbohydrate (mg/l)	12%	
Zinc (mg/l)	<7%	
Sulfide (mg/l)	<0.5%	
Copper (mg/l)	--	
OD_{205}	86%	
OD_{280}	48%	